

NASA TECHNICAL  
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NASA TM X-3425



NASA TM X-3425

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TWO-LAYER THERMAL BARRIER COATING  
FOR TURBINE AIRFOILS - FURNACE  
AND BURNER RIG TEST RESULTS

*Stephan Stecura*

*Lewis Research Center  
Cleveland, Ohio 44135*



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • SEPTEMBER 1976

1. Report No. <b>NASA TM X-3425</b>	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle <b>TWO-LAYER THERMAL BARRIER COATING FOR TURBINE AIRFOILS - FURNACE AND BURNER RIG TEST RESULTS</b>		5. Report Date <b>September 1976</b>	
		6. Performing Organization Code	
7. Author(s) <b>Stephan Stecura</b>	8. Performing Organization Report No. <b>E-8767</b>		
9. Performing Organization Name and Address <b>Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135</b>		10. Work Unit No. <b>505-01</b>	
		11. Contract or Grant No.	
12. Sponsoring Agency Name and Address <b>National Aeronautics and Space Administration Washington, D.C. 20546</b>		13. Type of Report and Period Covered <b>Technical Memorandum</b>	
		14. Sponsoring Agency Code	
15. Supplementary Notes			
16. Abstract <p>A simple, two-layer plasma-sprayed thermal barrier coating system was developed which has the potential for protecting high temperature air-cooled gas turbine components. Of those coatings initially examined, the most promising system consisted of a Ni-16Cr-6Al-0.6Y (in wt %) thermal barrier coating (about 0.005 to 0.010 cm thick) and a <math>ZrO_2</math>-12<math>Y_2O_3</math> (in wt %) thermal barrier coating (about 0.025 to 0.064 cm thick). This thermal barrier substantially lowered the metal temperature of an air-cooled airfoil. The coating withstood 3200 cycles (80 sec at <math>1280^0</math> C surface temperature) and 275 cycles (1 hr at <math>1490^0</math> C surface temperature) without cracking or spalling. No separation of the thermal barrier from the bond coating or the bond coating from the substrate was observed.</p>			
17. Key Words (Suggested by Author(s)) <b>Thermal barrier Oxide Alloy</b>		18. Distribution Statement <b>Unclassified - unlimited STAR Category 26</b>	
19. Security Classif. (of this report) <b>Unclassified</b>	20. Security Classif. (of this page) <b>Unclassified</b>	21. No. of Pages <b>19</b>	22. Price* <b>\$3.50</b>

\* For sale by the National Technical Information Service, Springfield, Virginia 22161

# TWO-LAYER THERMAL BARRIER COATING FOR TURBINE AIRFOILS - FURNACE AND BURNER RIG TEST RESULTS

by Stephan Stecura

Lewis Research Center

## SUMMARY

Adherence, thermal shock resistance, and resistance to cracking were evaluated for  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$ ,  $\text{ZrO}_2\text{-}3.4\text{MgO}$ , and  $\text{ZrO}_2\text{-}5.4\text{CaO}$  (all in wt % unless stated otherwise) thermal barriers on a variety of superalloys using Ni-16Cr-6Al-0.6Y alloy as a bond coat. The bond coating and thermal barrier coatings were plasma sprayed in air using argon as a plasma torch gas. The tests were conducted on coated coupon specimens in a cyclic furnace and on air-cooled coated turbine blades in a Mach 0.3 burner rig.

It was found that in the cyclic furnace testing  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  withstood 673 1-hour cycles at  $975^\circ\text{C}$  without cracking or spalling. The  $\text{ZrO}_2\text{-}3.4\text{MgO}$  coating cracked and spalled after 460 1-hour cycles at  $975^\circ\text{C}$ , and  $\text{ZrO}_2\text{-}5.4\text{CaO}$  cracked and spalled after 255 1-hour cycles at  $975^\circ\text{C}$ . The failures occurred in the oxide coating near the thermal barrier - bond coating interface.

The data from the Mach 0.3 burner rig tests on the  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  coated air-cooled J-75 turbine blades showed that underneath the coating the outer blade alloy surface temperatures are from  $140^\circ$  to  $190^\circ\text{C}$  lower than those for an uncoated blade exposed at the same burner rig conditions. As expected, the outer surface temperature of the thermal barrier coating is significantly higher than that for an uncoated blade. Also, in the cyclic burner rig tests of the air-cooled coated J-75 turbine blades, the  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  thermal barrier coating withstood 3200 cycles (80 sec at  $1280^\circ\text{C}$  surface temperature) and 275 cycles (1-hr at  $1490^\circ\text{C}$  surface temperature). Furthermore, the approximate 3.8 cm diameter flame produced very high thermal gradients on the surface of the thermal barrier coating (up to  $500^\circ/\text{cm}$  at  $1440^\circ\text{C}$ ) and through the thermal barrier coating (up to  $645^\circ$  over a 0.064 cm thickness at  $1490^\circ\text{C}$ ). These gradients did not cause any cracking or spalling of the  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  coating. The  $\text{ZrO}_2\text{-}3.4\text{MgO}$  coating did not crack or spall after 1010 cycles (80 sec at  $1180^\circ\text{C}$  surface temperature) while the  $\text{ZrO}_2\text{-}5.4\text{CaO}$  coating failed after 200 cycles (80 sec at  $1280^\circ\text{C}$  surface temperature). On all of the tested blades, the loss of coating thickness occurred in the hot zone. However, the  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  coating was the least affected.

## INTRODUCTION

Higher operating temperatures in advanced aircraft gas turbine engines have been achieved through a combination of advanced air cooling and higher use-temperature superalloys. Conventionally cast alloys are reaching the limits of compositional improvement, and directionally solidified alloys that do have significantly higher use potential are very expensive to cast. In addition, air cooling is also becoming more complex with many costly holes and expensive internal air distribution fixtures. Furthermore, an increased use of compressor bleed air introduces a growing penalty on engine performance. For these reasons, an alternate approach of insulating the airfoil surfaces from the hot gases has been pursued at NASA Lewis Research Center. It involves the use of thermal barrier coatings. Such thermal barrier coatings can reduce metal temperature, cooling air use, and/or costly cooling holes.

Such thermal barrier coatings have been under development for many years. The early work was aimed at minimizing heat transfer in regeneratively cooled rocket engines (refs. 1 to 3). Previous studies have also been made to apply such coatings to aircraft gas turbine components (refs. 4 and 5). Nichrome bond and calcia-stabilized zirconia thermal barrier coatings have been applied on air-cooled J-75 engine blades and vanes (ref. 6). It was reported that J-75 blades and vanes withstood 150 hours at leading-edge vane and blade steady-state metal temperatures up to  $647^{\circ}$  C and transient values as high as  $927^{\circ}$  C. Nijpjes (ref. 7) evaluated various bond and thermal barrier coatings. He found that Nimonic 115 alloy was a better bond coating than nichrome for calcia-stabilized zirconia. However, he reported that the adherence of the coating was still a major problem in the  $1200^{\circ}$  to  $1400^{\circ}$  C temperature range.

As part of a continuing NASA effort on thermal barrier coatings, a concept for a highly adherent two-layered thermal barrier was developed by this author. To explore this concept, a material scoping study was conducted to determine the coatings' adherences in cyclic furnace, cyclic burner torch rig, and Mach 0.3 burner rig tests. The purpose of this report is to present some of the initial results of this study. The effort was primarily focussed on a simple, two-layered plasma sprayed coating to minimize process and quality control problems. The inner coating layer on bond coating was a NiCrAlY alloy while the outer coating layer or thermal barrier coating was a stabilized zirconium dioxide.

## MATERIALS, APPARATUS, AND PROCEDURE

### Materials

The chemical compositions of the plasma spray powders of NiCrAlY (-200 to +325

mesh) and of zirconia stabilized with yttria, magnesia, or calcia (all -200 to +325 mesh) are presented in table I. The cast superalloy substrate materials were nickel-base alloys - B-1900 with Hf, directionally solidified (DS) MAR-M-200 with Hf, and conventionally cast MAR-M-200 with Hf. Cobalt alloy MAR-M-509 was also used. These materials were chosen because they are now or will soon be used in commercial aircraft engines. The analyzed compositions of these alloys are also reported in table I.

The original  $ZrO_2$ -12 $Y_2O_3$  powder as determined by X-ray diffraction analysis (XRDA) was found to be primarily cubic. (All compositions in wt % unless otherwise stated.) The  $ZrO_2$ -3.4MgO was also primarily cubic as determined by XRDA. Two types of  $ZrO_2$ -5.4CaO were used. One was originally 100 percent monoclinic (it was sprayed as a mixture of  $ZrO_2$  and  $CaCO_3$ ), but after spraying it was primarily cubic with some residual monoclinic phase present. The other  $ZrO_2$ -5.4CaO was pretreated and was cubic before and after spraying.

The alloy specimens used in furnace testing were 2.5 by 2.5 by 0.3 centimeter (cm) with all corners and edges rounded to about a 0.16-cm radius. In addition, available cast B-1900 (no hafnium (Hf) but otherwise the same composition as for B-1900 with Hf in table I) air-cooled turbine blades for a J-75 engine, previously aluminized, exposed in engine operation for 200 to 500 hours, and subsequently surface blasted with grit alumina, were used to evaluate the thermal barriers in Mach 0.3 burner rig tests.

#### Apparatus and Procedure

Plasma spray coating deposition. - Within 10 minutes after grit blasting cleaning the surface with alumina, the NiCrAlY bond coating was applied on test specimen using a hand-operated plasma spray torch. Operating with argon as a plasma gas, the power setting was 350 amperes and 34 volts for the plasma spray torch and specific nozzle selected. The thickness of the NiCrAlY bond coating was held between 0.003 and 0.008 cm. The bond coating was applied in air without using any additional cover gas or inert gas spray chamber. The zirconia coatings were also applied in air within 25 minutes of completing the NiCrAlY coating. Again, argon was used as the plasma gas and the torch operating conditions were 550 amperes and 38 volts. The specimens were coated with stabilized zirconia to approximately the following thicknesses: 0.025, 0.038, 0.051, or 0.064 cm. For spraying both the NiCrAlY and zirconia coatings, the torch-to-substrate spray distance was maintained at about 13 to 15 cm.

Cyclic furnace testing. - In the furnace cyclic tests the coated specimens were exposed to air at  $975^\circ C$  for 1 hour (specimens reached temperature in about 4 min) followed by furnace cooling to  $280^\circ C$  within 1 hour. The temperature of the coated specimens was measured by means of a platinum - platinum-13-percent-rhodium thermocouple. The thermocouple junction was positioned about 2 cm above the coated

specimens which were supported by rectangular platinum boats.

Cyclic Mach 0.3 burner rig testing. - In the Mach 0.3 burner rig (ref. 8) tests, coated J-75 blades were cooled by line-air at about a 14 gram per second flow. Those blades were held in a fixed position as shown in figure 1. The area of the hot zone was about  $4 \text{ cm}^2$  along the leading edge and somewhat larger on the pressure side of the trailing edge. The surface temperatures of the coated blades in the burner rig tests were measured by an optical disappearing filament pyrometer. The true temperature was established by means of a calibration curve. This calibration curve was obtained in the following manner. A 0.16-cm-diameter hole was drilled through the center of a 5.1 by 1.3 by 0.3 cm calibration specimen as is shown in figure 2. A platinum - platinum-13-percent-rhodium thermocouple was installed (about 0.007 cm from the surface) and the specimen coated with NiCrAlY and  $\text{ZrO}_2\text{-Y}_2\text{O}_3$  as previously described. The coated specimen was heated in a natural gas-oxygen torch rig to a desired temperature. Twenty to twenty-five minutes was allowed to reach the equilibrium condition. At that time, the thermocouple bulk temperature and the surface optical disappearing filament pyrometer temperature were recorded. The resultant calibration curve is shown in figure 3. The temperature measurement was considered to be accurate to about  $\pm 2$  percent at  $1320^\circ \text{C}$ . It is apparent from figure 3 that the pyrometer temperatures are not greatly different from the thermocouple temperatures. The temperature differences are so small because the recorded pyrometer temperatures are higher than normal due to the reflective light at the surface of the specimen and partly to the fact that the measurement of temperature with the pyrometer was done through the flame. The curve in figure 3 was extrapolated beyond  $1320^\circ \text{C}$ .

In the Mach 0.3 burner rig tests the substrate temperature in the coated blade was measured by a Chromel-Alumel thermocouple imbedded in the leading edge. The thermocouple junction was imbedded about 5.7 cm from the root of the blade and about 0.013 cm from the blade alloy outer surface.

The surface temperature of the coated blade was also measured by an Ircon pyrometer. These pyrometer measurements of the surface temperatures were particularly useful in determining the surface temperature gradients.

The external and internal surface temperatures of the uncoated blade were also measured. Here, one Chromel-Alumel thermocouple was imbedded in the external surface of the leading edge about 0.013 cm deep; the second thermocouple was 0.114 cm deep as measured from the outer metal surface of the uncoated blade or about 0.013 cm from the inside air-cooled surface of the blade. Both thermocouples were located about 5.7 cm from the root of the blade.

## RESULTS AND DISCUSSION

### Cyclic Furnace Testing

The adherence of the  $ZrO_2-12Y_2O_3$ ,  $ZrO_2-3.4MgO$ , or  $ZrO_2-5.4CaO$  thermal barrier coating to the NiCrAlY bond coating was first evaluated in cyclic furnace testing under relatively slow thermal transients. The thickness of bond coating as previously stated, was between 0.003 and 0.008 cm and that of thermal barrier coating was varied as desired from levels of 0.025 cm and above. The substrates used in this study were cast DS MAR-M-200 with Hf, MAR-M-200 with Hf, MAR-M-509, and B-1900 with Hf specimens.

The data in table II show that  $ZrO_2-12Y_2O_3$  was the most adherent, followed by  $ZrO_2-3.4MgO$ , partially stabilized  $ZrO_2-5.4CaO$  derived from the  $ZrO_2$  plus  $CaCO_3$  spray powders, and totally stabilized zirconia powder. The data in table II show that in the furnace tests conducted none of the  $ZrO_2-Y_2O_3$  thermal barrier coatings failed after many hundreds of 1-hour cycles (1-hr at temperature and 1-hr cooling) between  $975^\circ$  and  $280^\circ$  C. The  $ZrO_2-3.4MgO$  coating failed in less than 460 1-hour cycles, partially stabilized  $ZrO_2-5.4CaO$  in less than 255 1-hour cycles, and totally stabilized  $ZrO_2-5.4CaO$  in less than 87 1-hour cycles. Furthermore, it is evident from the data in this table that the nature of the substrates studied (substrates' coefficients of thermal expansion are between  $16 \times 10^{-6}/^\circ C$  and  $19 \times 10^{-6}/^\circ C$  at  $1000^\circ$  C) had probably little effect on the adherence or performance of the thermal barrier coating described in this report. The failures of the  $ZrO_2-MgO$  and  $ZrO_2-CaO$  thermal barrier coatings normally occurred within the oxide layer very close to the bond coating. Furthermore, the failures always started with the formation of a small, visible crack at one of the corners of the specimens and the crack propagated along the edges. It is believed that the stresses in those areas are the greatest. Therefore, the geometry of the specimens probably did have a significant influence on the life of the coating.

Typical microstructures of a  $ZrO_2-12Y_2O_3$  thermal barrier coating on DS MAR-M-200 with Hf before and after testing are shown in figures 4(a) and (b). It was apparent that after 673 1-hour cycles of  $975^\circ$  C no failure was observed. A comparison of microstructures in figures 4(a) and (b) indicates that the thickness of the bond coating after a 673-hour exposure probably did decrease somewhat. However, since the bond coating did vary in thickness from sample to sample, no definite estimate can be made as to the extent of the degradation. The micrograph in figure 4(b) shows that even after 673 hours at  $975^\circ$  C the sprayed NiCrAlY bond coating is still intact. In addition, no cracks in the oxide layer or any kind of bond coating - oxide layer separation was observed.

It should be remembered that in a gas turbine the bond coating probably would be

significantly below 975° C. In fact, the maximum temperature of the bond coating will probably be below 880° C.

#### Cyclic Mach 0.3 Burner Rig Testing - Short Cycles

The measured thicknesses in the NiCrAlY bond coating and stabilized zirconia thermal barrier coatings, manually plasma sprayed on the J-75 blades, varied by as much as  $\pm 0.004$  and  $\pm 0.008$  cm, respectively. Oxide surfaces on the blades were not polished, and no attempt was made to determine the effect of surface roughness on the life of the thermal barrier coatings. Tests were conducted at surface temperatures in the 1190° to 1540° C range.

The advantage of the thermal barrier coated blade over an uncoated blade is illustrated by the data in figure 5. The data in figure 5 were obtained on a  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  coated blade and on an uncoated blade. Figure 5 shows that at the highest constant Mach 0.3 burner rig flame temperature, even though the coated surface is at 1390° C, as measured by the disappearing filament optical pyrometer, the temperature at the thermal barrier - substrate interface is only 870° C. This is 140° C lower than the 1010° C surface temperature of the uncoated blade at the same burner rig conditions. Similar benefits were also achieved at lower Mach 0.3 burner rig flame temperatures as is also illustrated in figure 5. Furthermore, the interface temperatures in the coated blade are also significantly lower than the corresponding inner wall surface temperatures in the uncoated blade. Since thermal barrier coated, air-cooled engine components can be operated in higher gas temperature environments than the uncoated air-cooled components and still maintain lower metal temperatures (thereby extend airfoils' lives), significant engine efficiency improvements can be anticipated.

As previously shown in figure 1, the size of the hot zone on the coated J-75 blades in the Mach 0.3 burner rig was about 4 cm<sup>2</sup> along the leading edge and somewhat larger on the pressure side of the trailing edge. The maximum temperatures were always 250° to 400° C higher than on the trailing edge. This difference in temperature between the leading and trailing edge areas varies with the temperature at which the leading edge is maintained. The surface temperature gradient along the yttria-stabilized zirconia-coated heated leading edge was about 220°/cm at 1280° C, 440°/cm at 1440° C, and 500°/cm at 1540° C. The middle of the heated zone was the hottest area. All test temperatures (from 1190° to 1540° C surface temperatures) were reached within less than 30 seconds. The cooling to less than the 75° C thermal barrier - substrate interface temperature was accomplished within 20 seconds. These large surface temperature gradients along the surface and the rapid heating and cooling rates did not cause spalling or cracking of either the  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  or the  $\text{ZrO}_2\text{-}3.4\text{MgO}$  thermal barrier coatings.

The results presented in figures 6(a) ( $T_{\text{surface}} = 1280^{\circ} \text{ C}$ ) and (b) ( $T_{\text{surface}} = 1190^{\circ} \text{ C}$ ) show that the  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  system has significantly better erosion resistance

than the  $\text{ZrO}_2$ -3.4MgO system and better crack resistance than the  $\text{ZrO}_2$ -5.4CaO system. When the photomicrographs from the leading edge areas before and after cyclic exposure were compared, it was observed that in the hot zone the thickness of the  $\text{ZrO}_2$ -12 $\text{Y}_2\text{O}_3$  layer decreased by about 50 percent after 3200 cycles (80 sec at a  $1280^0$  C surface temperature). Little change in either bond or thermal barrier coating microstructures was observed when comparing nonheated parts of the blade and those in the heated zone as can be seen in figures 7(a) and (b). The microstructures in figures 7(a) and (b) show that the aluminide coating on these blades was not totally removed by the alumina grit blasting operation. However, the presence of the aluminide coating did not appear to influence thermal barrier coating performance.

The comparison of the coating thicknesses from the heated and nonheated areas of the  $\text{ZrO}_2$ -3.4MgO coated J-75 blade showed a greater loss in oxide layer thickness than was observed with the  $\text{ZrO}_2$ -12 $\text{Y}_2\text{O}_3$  coating. After 1010 cycles (80 sec at  $1190^0$  C surface temperature) the amount of  $\text{ZrO}_2$ -MgO lost was determined from the photomicrographs to be about the same as for the  $\text{ZrO}_2$ - $\text{Y}_2\text{O}_3$  after 3200 cycles (80 sec at  $1280^0$  C surface temperature). Otherwise, the microstructures of the  $\text{ZrO}_2$ -MgO system before and after testing were very similar to those shown in figures 7(a) and (b).

The data in figure 6(a) show that partially stabilized  $\text{ZrO}_2$ -5.4CaO coating failed after 200 cycles (80 sec at  $1280^0$  C surface temperature). Two blades were tested, and the results from both tests were about the same. The failures of the  $\text{ZrO}_2$ -5.4CaO thermal barrier coating occurred in the oxide coating near the thermal barrier-bond coating interface and thus are similar to the furnace results.

Consequently, the  $\text{ZrO}_2$ -12 $\text{Y}_2\text{O}_3$  thermal barrier coating appears to be the most promising barrier system in this study while the  $\text{ZrO}_2$ -5.4CaO system is the least promising.

### Mach 0.3 Burner Rig Life Testing

The data in figures 6(a) and (b) showed that the  $\text{ZrO}_2$ -12 $\text{Y}_2\text{O}_3$  thermal barrier coating is capable of withstanding several thousand short cycles to high temperatures without spalling or cracking. Therefore, an effort was also conducted to determine the endurance life of the  $\text{ZrO}_2$ - $\text{Y}_2\text{O}_3$  thermal barrier coating at various temperatures.

The tests were run on J-75 air-cooled blades coated with the NiCrAlY bond coating and the  $\text{ZrO}_2$ -12 $\text{Y}_2\text{O}_3$  thermal barrier coating. The thickness of the bond coating varied between 0.005 and 0.010 cm, while the thermal barrier thickness was nominally at 0.051 or 0.064 cm. These values were obtained by measuring the thermal barrier thickness at the same location on the blade. It is true that the thermal barrier coating thicknesses could have varied by as much as 0.010 cm at different locations on the blade.

The data in figure 8 show that at surface temperatures of  $1410^{\circ}$ ,  $1440^{\circ}$ , and  $1480^{\circ}$  C the  $ZrO_2-12Y_2O_3$  thermal barrier coatings withstood 246, 182, and 158 1-hour cycles, respectively. Each test was stopped on the basis of an arbitrary visual observation of thinning of the thermal barrier coating. When after testing some of the coated blades were sectioned and the oxide layer measured, the thermal barrier coatings were conservatively estimated to have lost 40 to 60 percent of the original oxide layer. For example, the micrograph of the coated blade after 182 cycles (1 hr at  $1440^{\circ}$  C surface temperature) is very representative of the microstructures encountered from the other tests (fig. 9). The thinning of the thermal barrier coating was primarily limited to the hottest part of the heated zone. This thinning could be due to the presence of a significant quantity of carbon in the Mach 0.3 burner rig flame. The Mach 0.3 burner rig was operated at very high fuel to air ratios in order to reach the desired temperatures.

The data in figure 8 also show that the thermal barrier coating provided significant reductions in substrate temperature. The resulting large thermal gradients through the thermal barrier coating did not cause any cracking in the coating. Only at the  $1540^{\circ}$  C surface temperature condition did the coating develop a very small vertical crack located inside the hot zone at the leading edge. It is believed that the crack was caused by the presence of a hot spot (between  $1650^{\circ}$  and  $1700^{\circ}$  C) which developed during the test. However, in a supplemental effort it was found that a J-75 blade coated with about 0.064-cm-thick  $ZrO_2-12Y_2O_3$  withstood 205 cycles (1 hr at about  $1540^{\circ}$  C surface temperature) in a natural gas - oxygen torch rig. In this test no significant deterioration of the coating was observed.

It was further found that by increasing the thickness of the thermal barrier coating from 0.051 to 0.064 cm the crack resistance and adherence of the oxide thermal barrier coating to bond coating were not affected. This can be seen by comparing the data from the  $1410^{\circ}$  C tests. The  $1410^{\circ}$  C test data in figures 8 and 10 show that no cracks or spalling was observed after 240 cycles (1-hr at surface temperature). Both tests were stopped after it was visually observed that about 50 percent of the oxide coating was lost. None of the samples failed through cracking or spalling. The temperature gradient through the 0.064-cm-thick thermal barrier coating increased to about  $650^{\circ}$  C (fig. 10) as compared to around  $500^{\circ}$  C for the 0.051-cm-thick coating (fig. 8). This large gradient did not cause any adverse effect. Furthermore, the lives of the 0.064-cm-thick thermal barrier coatings were longer than those for the 0.051-cm-thick thermal barrier coatings (figs. 8 and 10). Again, the life testing of the 0.064-cm-thick barrier coatings was stopped when the visual observations showed that there was a significant decrease in the thickness of the thermal barrier coatings. After tests, when the blades were sectioned and examined metallographically, it was found that about 50 percent of the oxide layer was still present.

## SUMMARY OF RESULTS

A program was conducted to examine the adherence, thermal shock resistance, and resistance to cracking of thermal barrier coatings of  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$ ,  $\text{ZrO}_2\text{-}3\text{.4MgO}$ , and  $\text{ZrO}_2\text{-}5\text{.4CaO}$  derived from either the  $\text{ZrO}_2$  and  $\text{CaCO}_3$  or the totally stabilized  $\text{ZrO}_2\text{-}5\text{.4CaO}$  spray powders. (All compositions are in wt % unless stated otherwise.) Thermal barrier coatings were evaluated on a variety of superalloys using the Ni-16Cr-6Al-0.6Y alloy as a bond coating. Cyclic furnace oxidation tests were conducted at  $975^\circ\text{C}$ . Mach 0.3 burner rig tests were conducted on air-cooled blades at surface temperatures of  $1190^\circ$ ,  $1280^\circ$ ,  $1410^\circ$ ,  $1440^\circ$ ,  $1480^\circ$ , and  $1540^\circ\text{C}$  in both 80 seconds and 1 hour at surface temperature exposure cycles.

The following results were obtained:

1. The  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  thermal barrier coating is the most resistant to erosion and cracking and is the most adherent of the three coatings studied. This coating did not fail after 673 furnace cycles at  $975^\circ\text{C}$ , but it seems that the bond coating did suffer some degradation. The  $\text{ZrO}_2\text{-Y}_2\text{O}_3$  coating did not fail by cracking or spalling in the Mach 0.3 burner rig after more than 200 cycles (1 hr at  $1410^\circ\text{C}$  surface temperature).
2. The  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  thermal barrier coatings (0.051 cm thick) on air-cooled blades lowered the external metal surface temperature from  $140^\circ$  to  $190^\circ\text{C}$  compared to an uncoated blade at the same Mach 0.3 burner rig conditions. Furthermore, as one might expect, the surface temperatures of the thermal barrier coating are from about  $240^\circ$  to  $380^\circ\text{C}$  higher than those of the uncoated blade at constant burner rig conditions. With thicker thermal barrier coatings these differences in external metal surface and thermal barrier coating surface temperatures will be even greater.
3. The thermal barrier oxide layer became thinner with time in the burner rig tests but no gross spalling was observed.

## CONCLUDING REMARKS

This study indicates that thermal barrier coatings have the potential to lower blade metal temperatures with no increase in cooling air mass flow. The decrease of up to  $190^\circ\text{C}$  observed in blade metal temperatures is significant in that the use temperature of superalloy blade materials has only increased at a rate of about  $10^\circ\text{C}$  per year. Also, while considerable material and process development, design consideration, and long time demonstration remain to be accomplished on the thermal barrier coatings,

they do offer a relatively straight forward way to exploit low cost state-of-the-art cast blade technology in advanced, high inlet temperature engines.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, June 17, 1976,  
505-01.

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TABLE I. - COMPOSITIONS OF SPRAY POWDERS AND SUPERALLOY SUBSTRATES

Element	Bond coating NiCrAlY	Composition, wt %						Cobalt-base substrate MAR-M-509
		Thermal barrier oxides			Nickel-base substrate			
		ZrO <sub>2</sub> -Y <sub>2</sub> O <sub>3</sub>	ZrO <sub>2</sub> -MgO	ZrO <sub>2</sub> -CaCO <sub>3</sub>	ZrO <sub>2</sub> -CaO <sup>a</sup>	B-1900 + Hf	DS MAR-M-200 + Hf	MAR-M-200 + Hf
Al	5.56	0.06	0.03	0.09	0.07	6.16	5.30	5.03
B	<.01	ND	<.01	<.01	<.01	.02	.02	<.01
C	ND	ND	ND	ND	.09	.16	.12	.60
Ca	<.01	.24	.14	3.86	3.82	ND	ND	ND
Co	.06	ND	ND	ND	ND	10.41	9.97	10.19
Cr	16.24	↓	↓	↓	↓	8.18	8.22	8.61
Cu	<.03					ND	<.01	<.10
Fe	.01	.16	.07	.13	.14	.14	.21	.35
Hf	<.01	2.00	2.00	2.00	2.00	1.19	2.25	1.97
Mg	ND	.06	2.00	.57	.60	ND	ND	ND
Mn	.01	ND	ND	ND	ND	<.01	<.01	<.01
Mo	<.01					5.77	ND	ND
Nb	<.01					.01	1.05	1.19
Ni	Major					Major	Major	10.46
O <sub>2</sub>	.02	↓		↓	↓	ND	ND	ND
P	<.01					ND	↓	↓
S	<.01					<.01	<.10	.20
Si	<.02	.23	.25	.19	.22	<.10	ND	.21
Ta	ND	ND	ND	ND	4.16	ND	ND	3.73
Ti	ND	.06	.13	.15	.13	ND	2.25	.17
W	.01	ND	ND	ND	ND	<.10	11.60	6.90
Y	.61	9.11	<.10	ND	ND	ND	ND	ND
Zr	ND	Major	Major	Major	.07	.06	.09	.49

<sup>a</sup>Totally stabilized calcia-zirconia powder as determined by X-ray diffraction analysis.<sup>b</sup>ND, not determined.

TABLE II. - CYCLIC FURNACE EVALUATION OF VARIOUS ZIRCONIA THERMAL  
BARRIER COATINGS ON Ni-16Cr-6Al-0.6Y BOND COATING TO 975° C

Alloy	Cycles to failure <sup>a</sup> - First visible crack, spall, etc.			
	ZrO <sub>2</sub> -12 Y <sub>2</sub> O <sub>3</sub>	ZrO <sub>2</sub> -3.4MgO	ZrO <sub>2</sub> -5.4CaO-P <sup>b</sup>	ZrO <sub>2</sub> -5.4CaO-T <sup>c</sup>
DS MAR-M-200 + Hf	<sup>d</sup> 673	460	255	78
MAR-M-200 + Hf	<sup>d</sup> 650	450	255	87
MAR-M-509	<sup>d</sup> 558	450	196	76
B-1900 + Hf	<sup>d</sup> 628	438	226	--

<sup>a</sup>Cycle, 1-hr at temperature and 1-hr to cool to 280° C.

<sup>b</sup>P, partially stabilized zirconia derived from ZrO<sub>2</sub> and CaCO<sub>3</sub> spray powders (cubic and monoclinic phases).

<sup>c</sup>T, totally stabilized zirconia derived from stabilized spray powder (cubic phase).

<sup>d</sup>No failure observed.

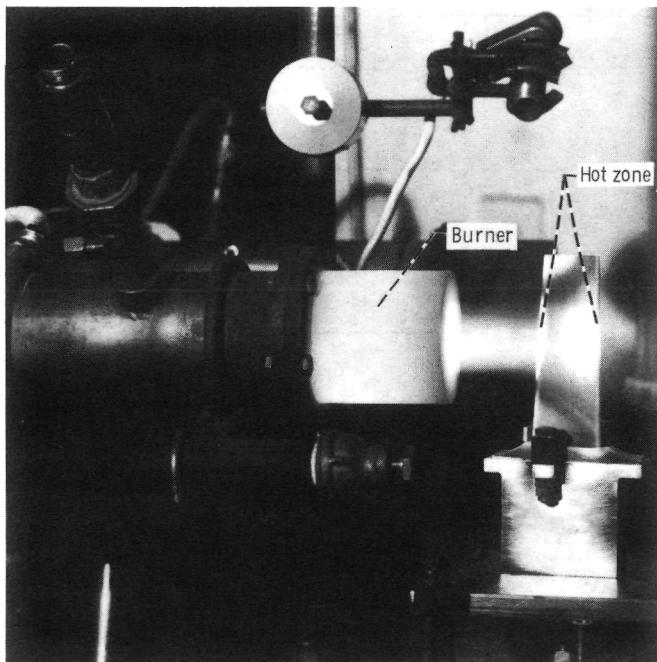


Figure 1. - Mach 0.3 combustion rig and J-75 blade coated with ZrO<sub>2</sub> - 12 weight percent Y<sub>2</sub>O<sub>3</sub> thermal barrier.

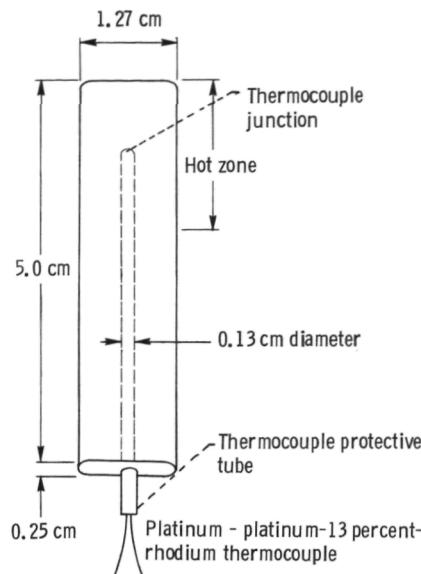


Figure 2. - Thermal barrier coated specimen with thermocouple used for temperature calibration.

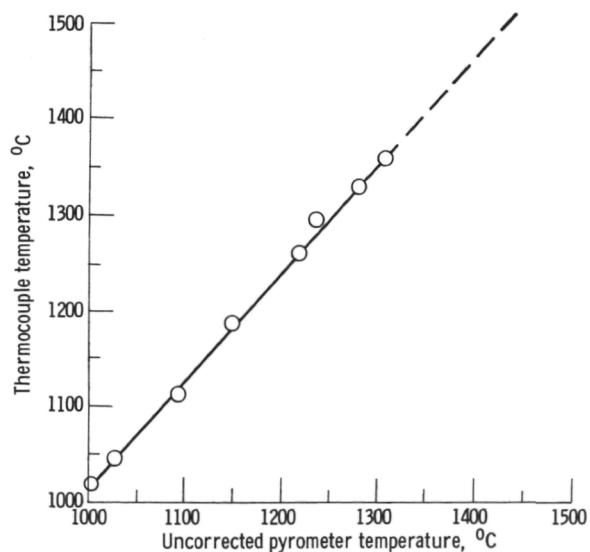
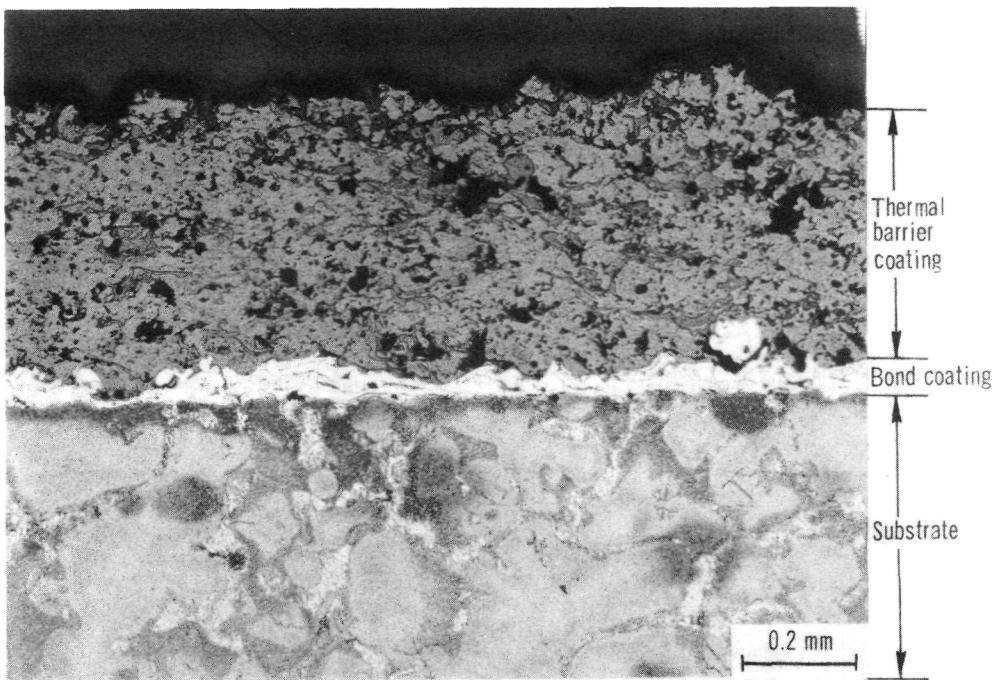
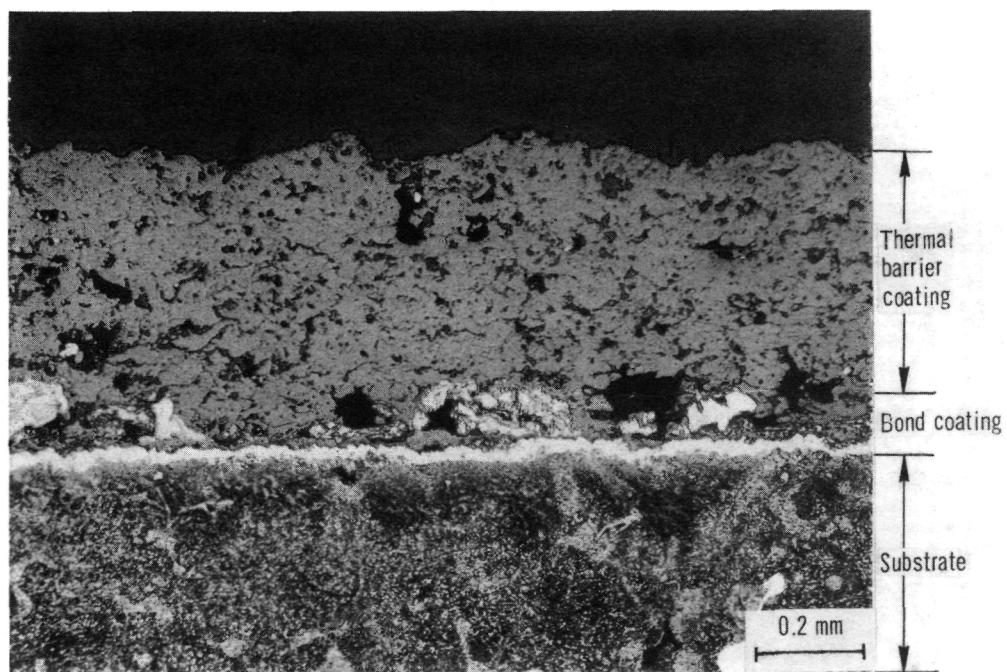


Figure 3. - Calibration curve for measuring thermal barrier coat surface temperature. Temperature accuracy,  $\pm 2$  percent.

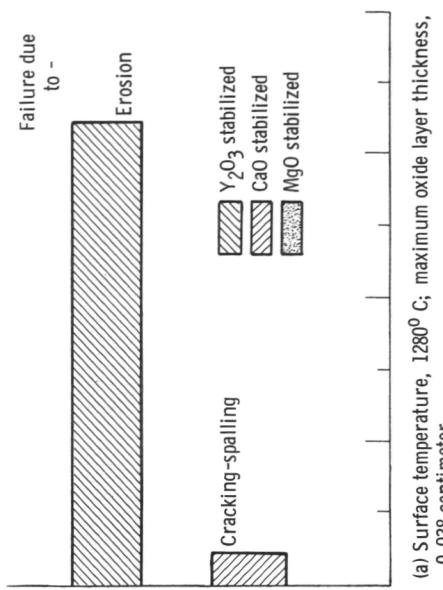


(a) Before testing.



(b) After testing for 673 cycles at 975°C. (Cycle, 4 min heat up, 60 min at temperature, and 60 min cooling.

Figure 4. - Light optical photomicrograph of DS MAR-M-200 with Hf coupon specimen coated with Ni-16Cr-6Al-0.6Y bond coating and  $\text{ZrO}_2\text{-}12\text{Y}_2\text{O}_3$  thermal barrier coating.



(a) Surface temperature, 1280°C; maximum oxide layer thickness, 0.038 centimeter.

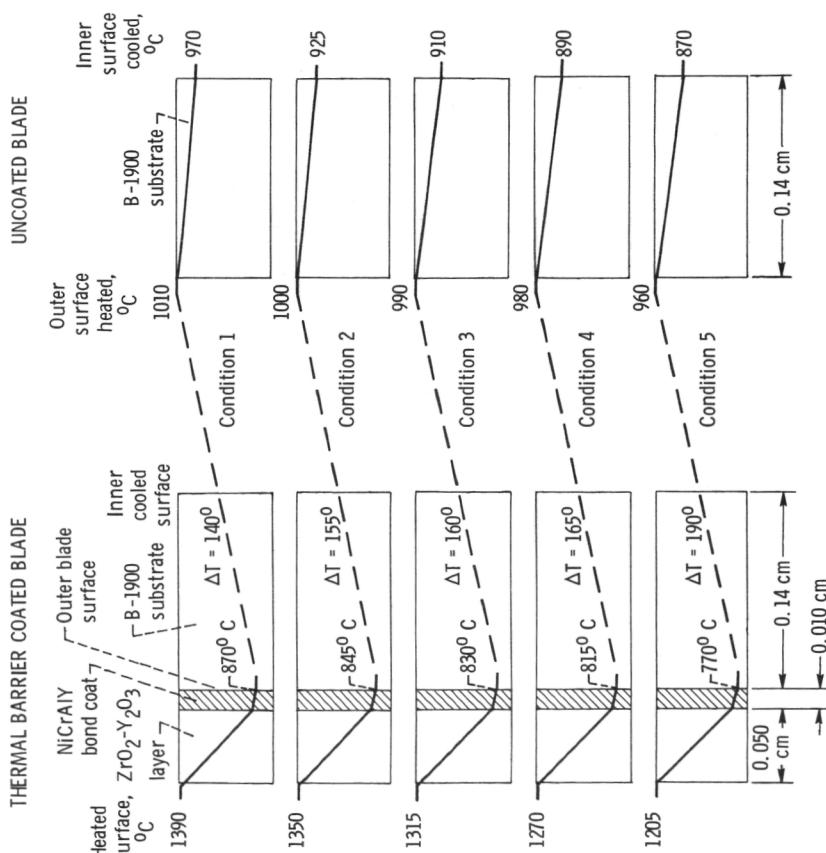
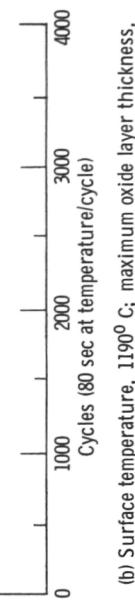
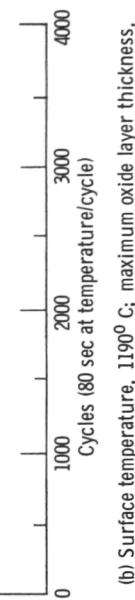


Figure 5. - Temperatures on cross sections of thermal barrier coated and uncoated J-75 blades at various Mach 0.3 burner rig conditions.

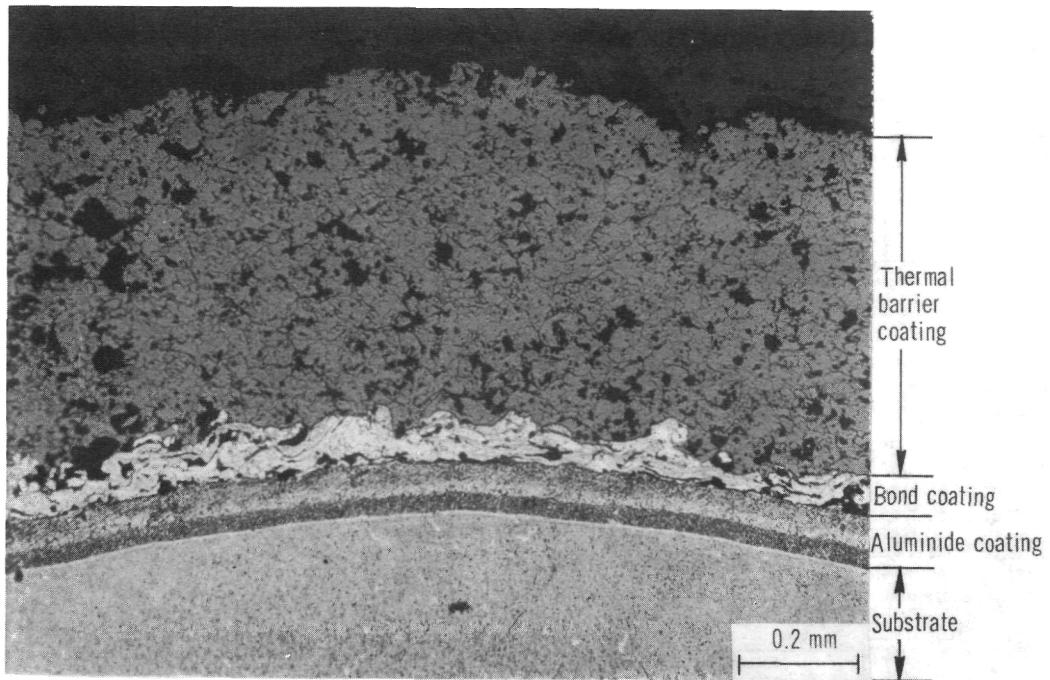


(a) Surface temperature, 1280°C; maximum oxide layer thickness, 0.028 centimeter.

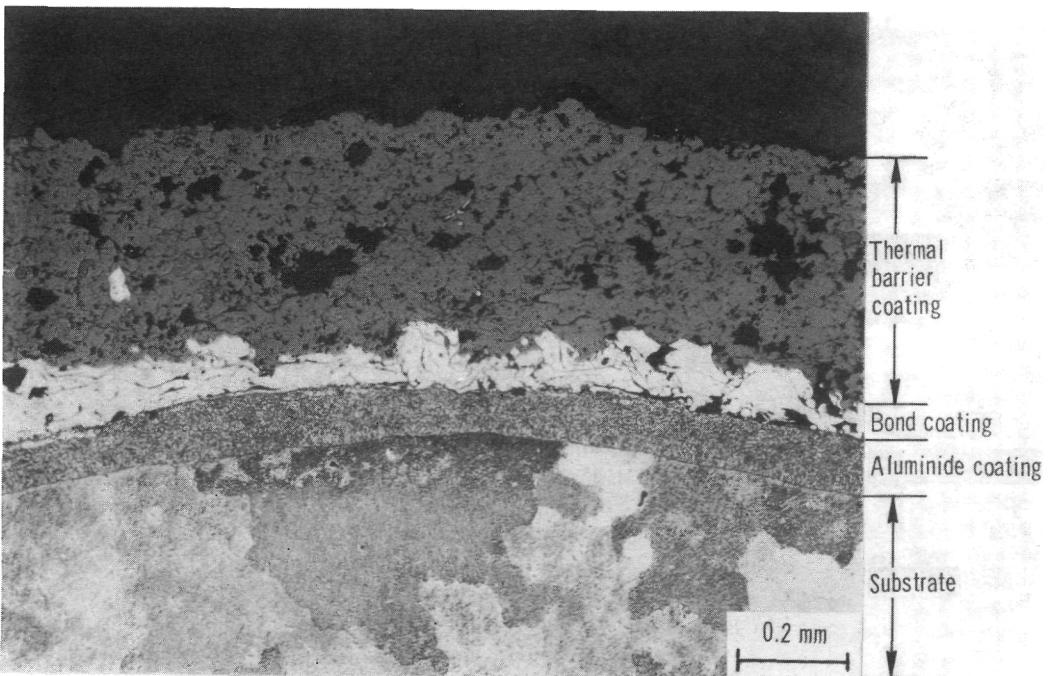


(b) Surface temperature, 1190°C; maximum oxide layer thickness, 0.028 centimeter.

Figure 6. - Cyclic performance of stabilized zirconia thermal barrier coatings on air-cooled J-75 turbine blades in Mach 0.3 burner rig. Substrate temperature, 915°C; failure determined on basis of visual observation of coating thickness loss by erosion.



(a) Before heating.



(b) After heating for 3200 cycles at 1280°C surface temperature and 915°C substrate temperature. (Cycle, 40 sec heat up, 80 sec at temperature, and 40 sec cooling.)

Figure 7. - Leading-edge area of air-cooled J-75 turbine blade (B-1900) coated with Ni-16Cr-6Al-0.6Y bond coating and  $ZrO_2$ -12 $Y_2O_3$  thermal barrier coating.

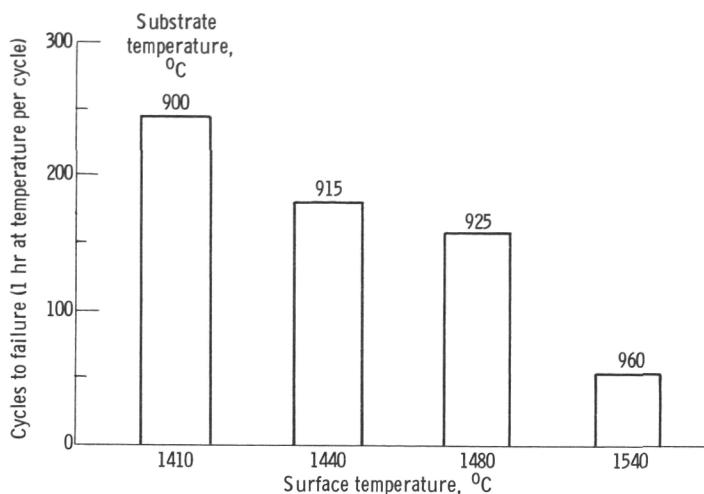


Figure 8. - Yttria-stabilized zirconia thermal barrier coating lives at high surface temperatures. Oxide thickness, 0.051 centimeter; failures determined on basis of visual observation of coat thickness loss (40 to 50 percent) by erosion.

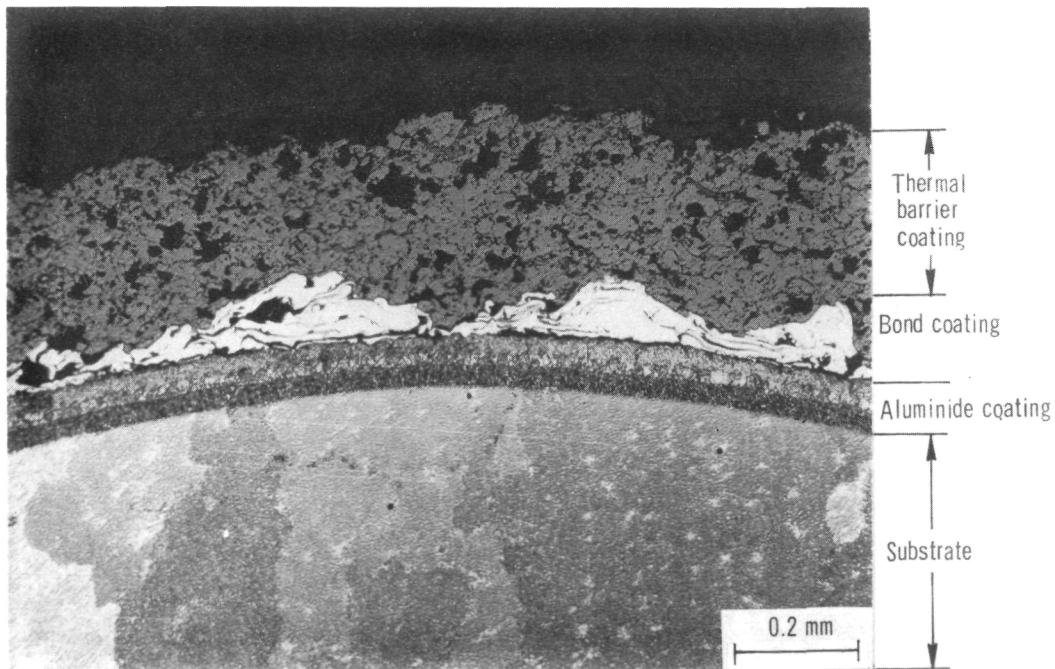


Figure 9. - Leading edge area of air-cooled J-75 turbine blade (B-1900) coated with Ni-16Cr-6Al-0.6Y bond coating and  $ZrO_2-12Y_2O_3$  thermal barrier coating after 182 cycles at  $1440^{\circ}\text{C}$  surface temperature and  $915^{\circ}\text{C}$  substrate temperature. (Cycle, 1-hr at temperature and 1 min cooling.)

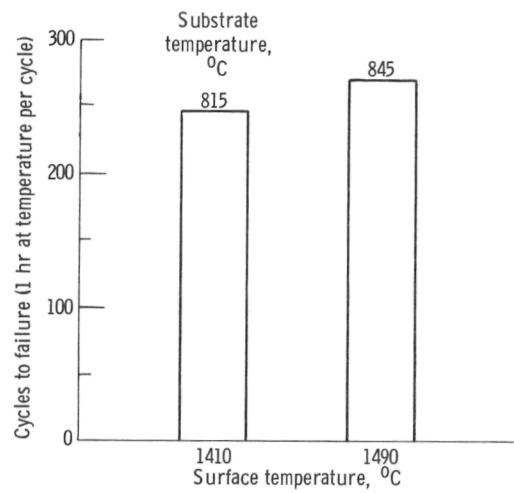


Figure 10. - Yttria-stabilized zirconia thermal barrier coating lives at high surface temperatures. Oxide thickness, 0.064 centimeter; failures determined on basis of visual observation of coat thickness loss (40 to 50 percent) by erosion.



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